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SAMPLING OF POLYCHLORINATED BIPHENYL AND VOLATILE ORGANIC EMISSIONS FROM A MOBILE PCB DESTRUCTION FACILITY

K.E. Curtis

As part of a Provisional Certificate of Approval for the operation of a Mobile PCB Destruction Unit (MPU-1), the Ontario Ministry of the Environment required that Ontario Hydro carry out an emission testing program. Concentrations of PCB and 14 specified volatile organic compounds were to be measured both before and after the emission control device, consisting of two carbon filters in series. A sampling protocol was designed and submitted to the Ministry for approval prior to the test program. Samples were collected on three consecutive days in December, 1987 and submitted for analyses. The analytical results, emission concentrations and discussion of these results are reported separately.

1.0 INTRODUCTION

Ontario Hydro possesses approximately 3 million litres of insulating oil that are contaminated with low levels of polychlorinated biphenyls (PCB) in excess of the 50 mg/kg limit that defines PCB waste/1/. In order to recover this oil, or to reduce its PCB content to an acceptable level before disposal, a process was developed in which the chlorine is stripped from the parent compounds by reaction with elemental sodium. The details of this process have been documented elsewhere/2,3/.

This technology has been developed to the stage where the process can be carried out in a mobile trailer that can be moved from site to site, rather than having to ship contaminated oil to a single fixed location for processing. The unit owned by Ontario Hydro is known as a Mobile PCB Destruction Unit and is designated as MPU-1. The details of this unit are also described elsewhere/1/.

job	file	date	report no.
33-254-343	837.29	June 3, 1988	88-145-K

89-02554/

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Prior to its use in Ontario, Ontario Hydro was required by the Ontario Ministry of the Environment (MOE) to apply for a Certificate of Approval for the operation of MPU-1. Part of the Provisional Certificate of Approval that was granted required that the emissions in the vent gases from the process be measured. This report describes the sampling program that was carried out to meet this requirement. The analysis of the collected samples and the resulting emission concentrations are described separately/4,5/.

2.0 THE SAMPLING PROGRAM

2.1 The Exhaust Gas System

The processing of a batch of oil consists of two basic stages. In the first stage, the oil is pumped through a degassifier into the reaction vessel. The second stage involves the actual destruction of the PCB by reaction with elemental sodium.

All of the resulting gases from the processing of a batch of oil, whether emitted during the filling stage or during the processing (destruction) stage, are vented through a single exhaust system. These emissions are controlled by passage through a series of two filters before being vented to the ambient atmosphere. The first filter is a 5 cm thick bed of granular activated charcoal. The second is a cartridge filter impregnated with activated charcoal. Collectively, these filters will be referred to as the emission control device throughout the rest of this report.

2.2 Proposed Sampling Protocol

The sampling protocol submitted to the MOE is shown in Appendix A and the associated equipment is shown in Figure 1. The procedures were based on NIOSH Method Nos P&CAM 178 (6) and P&CAM 244 (7) for vinyl chloride in air and PCB in air, respectively. It should be noted that the protocol proposed a total sample volume of about 5 litres for both types of samples. In retrospect, the recommended sample volume of 50 litres for PCB sampling should have been used. This was because it was later found that a detection limit of 450 ng/m3 for the PCB emissions was desired, rather than the method's lower limit of 0.01 mg/m3. This difference of a factor of about 200 also caused the analysis protocol to be changed in an attempt to meet this lower limit/4/.

The MOE made two comments in response to this submission which resulted in the following changes:

(i) Concern was expressed over potential adverse effects that could be caused by high levels of water vapour in the gas stream. It is known that high humidity severely decreases the capacity of activated carbon to trap organic vapours, especially if condensation actually occurs in the absorption tube/6/. Therefore, it was agreed to carry out several moisture determinations prior to the actual tests.

(ii) The use of individual totalizing flow meters in the place of the four rotameters was recommended. This was not accepted because of their high cost and the very long delivery time quoted by the supplier. Instead, it was agreed to recalibrate the rotameters and the dry gas meter following the tests.

Two other deviations from the proposed sampling program were necessary. Firstly, operational constraints prevented the three tests as described in the protocol (blank run, low PCB content and highest PCB content) from being carried out. Instead, three batches of the same oil with the highest PCB content were used. The other change involved the collection of separate sets of samples during the two stages of the process, one during the filling stage and another during the processing stage. This was necessary so as to not exceed the maximum sample volume of 5 L allowed for trapping the organic emissions/6/, while maintaining a reasonable sampling rate.

2.3 Sampling Equipment

The various components of the sampling trains used are shown in Figure 1. Four samples were collected in each test, two at the inlet to the control device and two at its outlet. For each set of two samples, one was for PCB and the other for volatile organic compounds. The components of each sampling train consisted of:

(a) Two absorption tubes in series. The inlet to the first was inserted directly into the effluent stream and Teflon tubing was used to join the two tubes. Activated charcoal was used as the absorbent for volatile organics and Florisil was used for PCB collection.

(b) A calibrated Matheson Model 7631T-601 rotameter set for a flow of 50 mL/min, joined to the outlet of the second absorption tube with Tygon tubing.

(c) A diaphragm vacuum pump equipped with a regulating valve open to the atmosphere at its inlet. This allowed the flow through each of the four rotameters to be controlled while minimizing the pressure drop across each.

A calibrated Rockwell Model RC-M-230 dry gas meter followed by a Dwyer 50 L/min rotameter were plumbed into the exhaust gas system downstream from the sampling points at the control device outlet. These were used to measure the total volume of vent gas emitted during each test and, where applicable, to indicate the instantaneous flow rate of these emissions.

2.4 Calibration of Flow Measuring Devices

A Tylan 360 Series mass flow meter, equipped with a read-out box and a series of mass flow sensors with various ranges, was used as the primary calibration device. Each of the flow sensors had been factory calibrated to indicated the mass flow of air at 25°C and 101.3 kPa, and has been periodically checked against bubble meters since that time. The two flow sensors used for this work were the Model FM 360 4F with a range of 0 to 200 cm3/min and the Model FM 362 3F with a range of 0 to 100 L/min.

The four rotameters used were designated as follows for the four sampling systems:

A. Volatile organics at control device inlet,
B. PCB at control device inlet,
C. Volatile organics at control device outlet and
D. PCB at control device outlet.

To calibrate each rotameter, the 0-200 cm3/min flow sensor was plumbed into the line between the outlet of the rotameter under test and the pump (see Figure 1 for arrangement of rotameters and the pump). Also, a set of two Florisil tubes was placed in the inlet line to that rotameter. All four rotameters were then run at settings approximating flows of 50 cm3/min, as indicated by the typical flow calibration graphs supplied with the units. The flow setting on the rotameter being calibrated was then set to give a reading of 50 cm3/min on the mass flow meter. This procedure was repeated several times until settings had been established to give a flow of 50 cm3/min through all four rotameters.

For calibration of the dry gas meter, the 0-100 L/min flow sensor was installed between a source of compressed air and the inlet to the meter. The air flow rate was then adjusted to give various flows, as indicated by the mass flow meter, of between 10 and 100 L/min. Each corresponding flow rate recorded by the dry gas meter

was determined by measuring the time taken for the meter to indicate that a set volume of air had passed through it, usually between 2 to 10 m3. This volume was corrected to 25°C and divided by the time in minutes to give a flow rate in L/min. A correction factor was then calculated by comparing this value to that indicated by the mass flow meter, allowing a calibration graph to be constructed over the range of flows tested.

These calibrations were carried out both before and following the test program. As will be discussed in the results section of this report, some of the calibration data recorded before the test program were invalidated by a MPU-1 system malfunction. This occurred during the first attempted test when oil entered the vent gas system and was drawn into the two rotameters preceding the control device, as well as the dry test meter.

2.5 Moisture Measurements

The moisture content of the vent gas upstream of the control device was determined both before and following the test program. Separate measurements were carried out during the filling stage and the processing stage for each determination.

For each measurement, a preweighed polyethylene absorption tube containing about 20 g of 6-16 mesh indicating silica was connected to one of the sampling points at the inlet to the control device. A Matheson Model 7631H-603 rotameter and a small vacuum pump were used to draw vent gas through the tube at a flow rate of 1 L/min for the duration of the measurement. The tube and its contents were then weighed again and any weight increase was assumed to be due to trapped water vapour. This assumption gave a conservative result in that the moisture would actually be lower than the measured value if any material other than water vapour had been trapped on the silica gel. Results were calculated in both mg/L and relative humidity at the gas temperature recorded during each measurement.

2.6 Sample Collection

Six runs were carried out during the test program, three during the filling stage and three during the processing stage of the PCB destruction process, representing the treatment of three complete batches of oil.

As described earlier in Section 2.3, four samples were collected during each of the six emission tests. For reporting purposes, they have been identified as PCB Inlet, PCB Outlet, Organics

Inlet and Organics Outlet.

Table I gives some of the more pertinent operating data that were recorded during the test program. It is interesting to note that the total volume of vent gas emitted during the filling stage was much greater than that released during the corresponding processing stage for each batch of oil treated.

It should be noted that the first attempt to sample the emissions was carried out on December 8, rather than December 15, 1987 as shown in Table I. However, that test was aborted after only two hours due to a process malfunction that caused the vent gas system to be swamped with oil. Unfortunately this oil was drawn into the two sampling systems upstream of the control device, including the rotameters. The two other sampling systems following the control device were switched off in time to prevent the oil from reaching those rotameters, but a considerable quantity was blown into the dry gas meter.

The two contaminated rotameters and the dry gas meter were subsequently disassembled, cleaned with various solvents and flushed with air for several days. However, there was insufficient time available before the test program to check the calibrations of these devices and they were therefore used as if the existing calibration data were still valid. All of the flow measuring devices were then recalibrated at the conclusion of the test program.

TABLE I

Operating Data for the MPU-1 and the Vent Gas Sampling System During Testing of the Emissions

	Emission Test Number					
	1	2	3	4	5	6
Date	Dec 15	Dec 15	Dec 16	Dec 16	Dec 17	Dec 17
Activity	Fill	Process	Fill	Process	Fill	Process
Volume of oil (L)	1576	-	1441	-	1510	-
PCB content (mg/kg)	3040	-	3000	-	2700	-
Volume of vent gas emitted (m3)	9.35	1.08	13.62	1.01	9.26	1.20
Volume of vent gas sampled (L)						
PCB :						
Inlet	5.20	8.66	8.74	6.88	5.47	3.34
Outlet	3.76	6.28	6.34	4.98	4.00	2.42
Organics :						
Inlet	4.14	6.90	6.97	5.48	4.36	2.67
Outlet	3.76	6.28	6.34	4.98	4.00	2.42

3.0 RESULTS AND DISCUSSION

3.1 Calibration of Flow Measuring Devices

3.1.1 Sampling System Rotameters

The rotameter calibrations done both before and after the test program are shown in Table II.

TABLE II

Calibration of Sampling System Rotameters

Rotameter Identification	Scale Settings for flow of 50 cm3/min		Flow rate at these settings after the test program (cm3/min)
	Glass ball	Steel ball	
A.	111	57	55
B.	106.5	58.5	69
C.	108.5	47.5	50
D.	105.5	57	50

These results confirmed that the two rotameters used at the control device outlet were unaffected by the system malfunction. However, the calibrations of the two that had been contaminated with oil were significantly altered. Since it was not known if these changes had occurred gradually over the time of the test program or were in effect for the whole period, the latter had to be assumed. Consequently, the flow rates shown in the last column of Table II were used to calculate the volumes of vent gas sampled during each test, as listed in Table I.

3.1.2 Dry Gas Meter

The data from the dry gas meter calibrations are shown in Appendix B, along with tables of correction factors and a graph of these factors versus flow rate.

In summary, correction factors ranging from about 1.03 at 11 L/min to 1.11 at 90 L/min were found during the initial calibration. After the test program (and contamination of the meter with oil), the range of these factors was again 1.03 at 11 L/min but as high as 1.20 at 84 L/min. The oil contamination had most significantly altered the performance of the meter at high flow rates, as might be expected. There was no significant effect at flow rates below about 40 L/min.

High vent gas flow rates of about 100 L/min or more were encountered only during the three filling operation test runs

(Tests No 1, 3 and 5). Since the calibration data covered only flows of up to about 90 L/min, a correction factor of 1.2 was applied to all these recorded dry gas meter volumes. This could have resulted in these volumes being underestimated. However, this would affect only the calculated total quantity of the emissions, not their concentrations in the vent gas.

All other volumes recorded during the tests were corrected using the appropriate factors and are considered to be accurate within the normal error range of a dry gas meter.

3.2 Moisture Measurements

The results from the four moisture determinations carried out are shown in Table III. The levels were highest during the processing stage of the procedure, most of which was probably released during the addition of "kill water" to the reaction tank. Even then, conditions did not exist that could cause condensation in the sampling tubes during the test program. Consequently, as far as the moisture content of the vent gas is concerned, there is no reason to doubt the collection efficiencies of the absorption tubes.

TABLE III

Moisture Content of MPU-1 Vent Gas

	Date	Time (min)	Moisture (mg/L)	Vent gas temperature	Relative humidity
Filling	Nov 27	96	3.75	23°C	18 %
Processing	Nov 27	55	19.1	24°C	88 %
Filling	Dec 17	107	1.78	21°C	9 %
Processing	Dec 18	235	7.49	15°C	58 %

As mentioned in Section 2.5, the procedure used for these measurements assumed all material trapped by the silica gel absorption tube was water vapour. In fact, observations made during the test program (and confirmed during subsequent analysis of the absorption tubes) suggest that some of this could well have been due to a fine oil mist present in the vent gas. This would mean that the actual relative humidities of the gas were actually lower than those reported in Table III.

3.3 Sample Collection

Other than the discovery of the presence of a fine oil mist in the vent gas preceding the control device, no other unusual observations or system malfunctions were noted during the sample collection period of December 15 to December 17, 1987.

The site was visited by Messers H. Corinthios and G. Wong from the Ontario Ministry of the Environment during the sample collection program. Other than confirming the need to recalibrate the flow measuring devices at the conclusion of the program, no significant comment was made.

The sampling program generated six sets of four samples, 12 for PCB analysis and 12 for analysis of volatile organics. Since each sample consisted of a first and a second absorption tube, this represented a total of 60 tubes, including a blank of each type for each test run. Each tube itself consists of a front and a back section that are analyzed separately, giving a potential for 60 PCB and 60 volatile organics analyses. However, the analytical protocol required the second absorption tube from a sample to be analyzed only if a significant quantity of the analyte was found in the back section of the first absorption tube. Consequently, the actual number of analyses required was somewhat less than the above numbers.

3.4 Analysis of Samples

A report detailing the methodolgy used to analyze the collected samples and the analytical data obtained is being prepared by Dr. R.E. Massey of the Chemical Research Department/4/.

3.5 Emission Concentrations of PCB and Volatile Organics

The data contained in this report has been combined with the analytical results from Dr. Massey to produce emission concentrations for each of the analytes. These results, along with other data such as removal efficiencies of the control device and analysis of the feed oils are contained in a report being prepared by Dr. K.E. Curtis of the Chemical Research Department/5/.

4.0 SUMMARY AND CONCLUSIONS

The vent gas emissions from the MPU-1 PCB destruction unit have been sampled during the processing of three batches of contaminated oil.

Apart from an MPU-1 system malfunction during the initial attempt to collect samples, the program was completed without any major mishaps. However, the presence of a fine oil mist in the vent gas prior to the control device has caused some problems with sample collection efficiency, analysis and the performance of the control device itself/5/.

Future work of this type should make use of totalizing volume meters for measuring the volumes of vent gas sampled, as recommended by the Ontario Ministry of the Environment. Also, the PCB sample trains should be run at much higher flow rates to allow the required detection limit of 450 ng/m3 to be more easily achieved.

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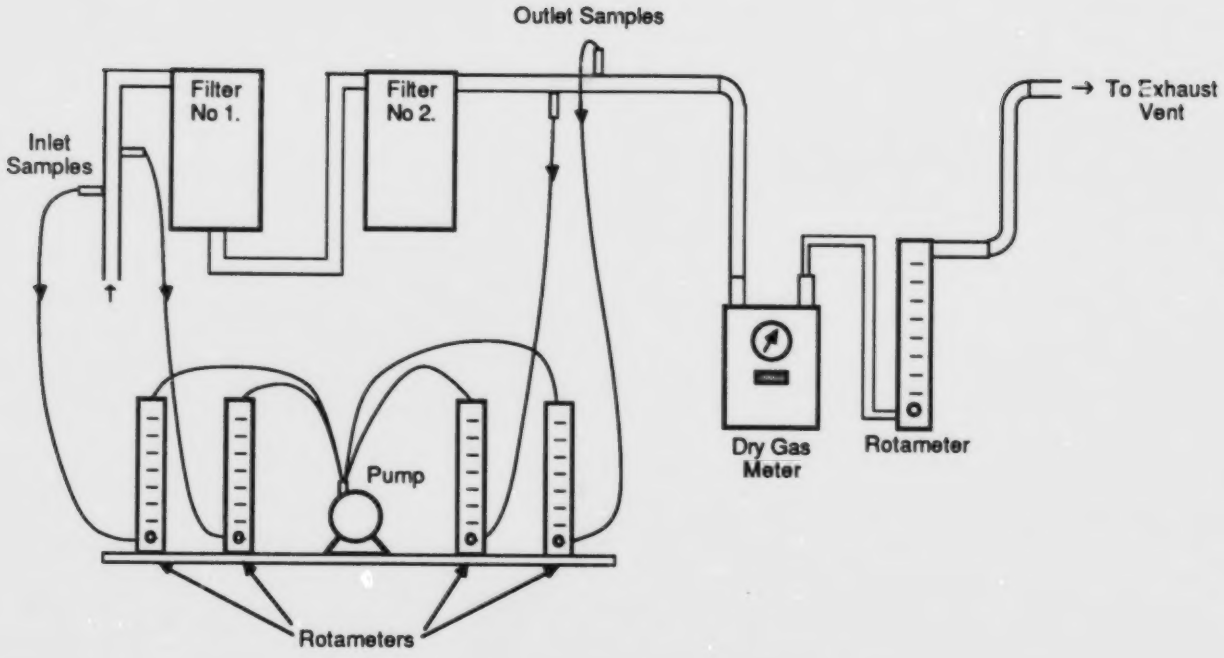

FIGURE 1

SAMPLING SYSTEM USED DURING MPU-1 EMISSION TESTING

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APPENDIX A

Sampling Protocol for Emission Testing of the MPU-1 Mobile PCB Destruction Facility Watse Management System

1. A schematic drawing of the MPU-1 exhaust system, the charcoal filters and the sampling apparatus is given in Figure 1.

2. Three tests will be run:

   (a) a blank test with the system charged with oil and nitrogen but with no sodium added,

   (b) a PCB destruction run with the system containing oil with the highest PCB content, and

   (c) a PCB destruction run with any other batch of contaminated oil.

3. During each test, the total effluent from the system will be passed through a calibrated dry gas meter and rotameter. This will allow the volume of gas emitted and its instantaneous flow rate to be measured during the test.

4. Four samples will be collected during each test, two at the inlet to the charcoal filters and two at their outlet.

   The two samples at the inlet will be collected on :

   (a) two SKC Florisil adsorption tubes in series for collection of PCBs, and

   (b) two Supleco ORBO-32 Charcoal adsorption tubes in series for collection of the other organic species of interest.

   The same combination of adsorption tubes will also be used to sample the outlet from the charcoal filters.

5. For each sample, the first adsorption tube in the series will be inserted directly into the effluent stream via a Swagelok fitting. Thus it will also act as the sample probe. The second tube in the series will be connected to the first with Teflon tubing. Polyethylene will then be used to connect the tubes to one of four calibrated rotameters and the pump.

6. Sample collection will be carried out to coincide with the full exhaust cycle of the test. Each sample flow rate will be controlled at between 30 to 50 mL/min so that the total volume of exhaust gas sampled does not exceed 5 liters.

7. Immediately following cessation of sampling, each of the four adsorption tubes will be capped and placed in a cooler containing dry ice. They will then be transported to the laboratory and stored in a freezer at - 20°C until they are analyzed.

APPENDIX B

Data from Calibrations of Flow Measuring Devices Before and and After the Sampling Program

DGMV Calibration Sept/87

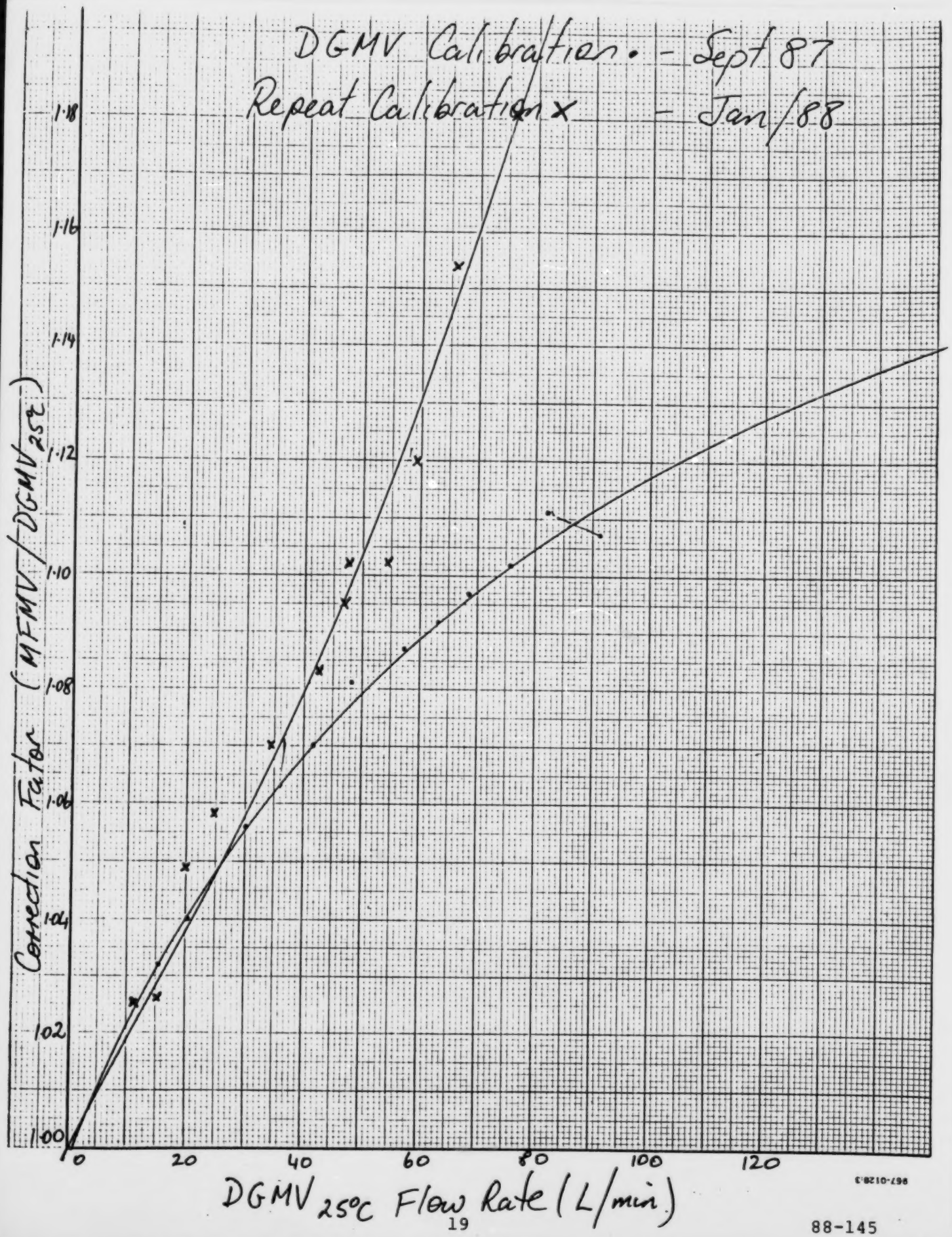
MFM (L/mm)	Time (min)	∴ MFMV (L)	DGMV (L)	Temp (K)	$DGMV_{25°C}$ (L)	$DGMV_{25}$ L/mm	MFMV / $DGMV_{25°C}$
11.3	202	2283	2210	296	2225	11.0	1.026
15.7	168	2638	2540	296	2557	15.2	1.032
21.0	155	3255	3110	296	3131	20.2	1.040
31.8	118	3752	3530	296	3554	30.1	1.056
44.5	191	8500	7890	296	7943	41.6	1.070
51.9	63.3	3286	3020	296	3040	48.0	1.081
62.2	58.6	3645	3330	296	3353	57.2	1.087
69.0	52.1	3595	3260	295	3293	63.2	1.092
74.9	46.9	3513	3170	295	3202	68.3	1.097
83.5	40.0	3340	3000	295	3030	75.8	1.102
91.1	36.7	3343	2980	295	3010	82.0	1.111
100.8	31.4	3165	2840	296	2859	91.1	1.107

Before tests

Repeat DGMV Calibration After Tests

Jan/88

MFM (L/min)	Time (min)	∴ MFMV (L)	DGMV (L)	Temp (K)	$DGMV_{25C}$ (L)	$DGMV_{25}$ (L/min)	MFMV / DGMV
11.9	191	2273	2210	297	2217	11.6	1.025
15.5	182	2821	2740	297	2749	15.1	1.026
21.0	220	4620	4390	297	4405	20.0	1.049
26.2	182	4768	4490	297	4505	24.8	1.058
36.7	117	4294	4000	297	4013	34.3	1.070
46.0	151	6946	6390	297	6412	42.5	1.083
51.3	109	5592	5090	297	5107	469	1.095
52.5	110	5775	5160	296	5195	47.2	1.112
60.2	146	6983	6240	296	6282	54.2	1.112
66.3	147	9746	8640	296	8698	59.2	1.120
76.2	78.2	5960	5130	296	5165	66.0	1.154
91.0	67.6	6152	5190	297	5207	77.0	1.181
101.1	81.7	8260	6850	297	6873	84.1	1.202


DGMV Calibration • - Sept 87
Repeat Calibration × - Jan/88
Correction Factor (MFMV/DGMV25°C)
1.18
1.16
1.14
1.12
1.10
1.08
1.06
1.04
1.02
1.00
0
20
40
60
80
100
120
DGMV 25°C Flow Rate (L/min)
867-0128-3

88-145

DGMV Calibrations Before Tests Date: Aug 31/87

START TIME	MFM Reading (L/min)	DGMV Start	DGMV Stop	DGM Temp In	DGM Temp Out	Time of test Hour	Time of test Min
9.20	11.3	29.740		23	23		
11.25	11.2 - 11.3	2210		✓	✓		202
	11.3 - 11.4		31.950	✓	23°C	3h	42 min
1.25 pm	15.6	31.960		23	23		
2.30 pm	15.7	2540		✓	✓		168
	15.7 - 15.8		34.500	✓	23°C	2h	48
Sept 1							
8.05	20.9 - 21.0 ✓	34.580	3110	23	23		155
10.40	21.0		37.690	✓	23°C	2h	35
10.55	31.8	37.720	3530	23	23		118
	✓		41.250	✓	23°C	1h	58 m
1.10 pm	44.4	41.280		23	23		
	44.5	7890		✓	✓		191
	44.5		49.170	23	22	3h	11 min

20

88-145

DGMV Calibrations

Date: Sept 2/87

START TIME	MFM Reading (L/min)	DGMV Start	DGMV Stop	DGM Temp In	DGM Temp Out	Time of test Hour	Time of test Min
8·10	51·9 ✓	49·280 3020	52·300	23 ✓ 23	23 ✓	1 h	3:20 63·3
9·25	~~52·36~~ 62·1 62·1-62·2	52·340 3330	55·670	23 23	22½ 22	0	58:34 58·6
10·35	69·0 ✓	55·720 3260	58·980	23 22	22 22 22°C	0	52:07 52·1
11·35	74·8 74·9+	59·040 3170	62·210	22 22	22 ✓	0	46:52 46·9
12·35	83·5 ✓	62·290 3000	65·290	22 ✓	22 ✓	0	40:00 40.0
1:30	91·2-91·1 91·1	65·400 2980	68·380	22	22 22°C	0	36:43 36.7
2·30	100·8 ✓ 100·8	68·510 28 40	71·350	23 ✓ 22	22 23		31:24 31·4

After Tests

DGMV Calibrations

Date: Jan 4/88

START TIME.	MFM Reading (L/min)	DGMV Start	DGMV Stop	DGM Temp In	DGM Temp Out	Time of test Hour	Time of test Min
8.20 10.30	11.8 11.9 (11.9)	15.820 2210 L	18.030	24 ✓ ✓	24 ✓ (24) ✓	3h	11 (191)
12.10pm	15.6 ✓ 15.5 15.5/4	18.060 2740	20.800	24 ✓	24 ✓ [24°C]	3	02 (182)
Jan 5 8.30	20.9 21.1/6 21.0	20.910 (4390)	25.300	24 ✓ ✓	24 23 (24°C)	3	(220) 40
12.35	26.2 ✓ 26.1-.2	25.340 [4490]	29.830	24 ✓ ✓	23 23-24 [24°C]	3	02 182
Jan 6 8.15	36.6 36.7 ✓	29.980 (4000)	33.980	24 ✓	24 ✓ (24°C)	1	57 (117)
10.25	46.0 ✓	34.030 *	(6390) 40.420	24 ✓	(24)	2	31 151

DGMV Calibrations

Date: Jan 6 (contd)

START TIME	MFM Reading (L/min)	DGMV Start	DGMV Stop	DGM Temp In	DGM Temp Out	Time of test Hour	Time of test Min	
1·20 pm	51·4 ~~51·3~~ (51·3)	40·470 (5090)	45·560	24	24 (24)	1	49	(109)
Jan 7/88 8·25	~~52·50~~ ✓ 52·5	45·670	5160L 50·830	24 23	23 (23)	1	50	(110)
10·30 11 10	60·1 / ·2 60·2 (60·2)	50·880 (6240)	57·120	23 ✓ ✓	23 ✓ (23°C)	1	56	116
12·45 pm ≃ 1.30	(66·3) ✓ 66·4	57·160 (8640)	65·800	23 ✓ 24	23 ✓ (23)	2	27	147
Jan 8/88 8·10	~~65·920~~ 76·1 76·2 + (76·2)	65·920	(5130) 71·050	24 23 ✓	23 ✓ ✓ (23)	1L	18:14	(98·2)
88-145 9·40	91·0 ✓ (91·0)	71·160 5190	76·350	23½ 24	23½ ~~24~~ (23½)	1	07:38	67·6
11·00	101 101·1 (101·1)	76·490 6850	83·340	24 ✓	23 24 ✓ (24)	1	21·40	(81·7)